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AN EVALUATION OF A BAKEOUT PROCEDURE FOR SMALL GLASS ULTRAHIGH VACUUM SYSTEMS

F. Steinrisser

REPORT R-299 JUNE, 1966

This work was supported in part by the Joint Services Electronics Program (U. S. Army, U. S. Navy, and U. S. Air Force) under Contract No. DA 28 043 AMC 00073(E), and in part by the National Aeronautics and Space Administration under Grant NASA NsG-376.

AN EVALUATION OF A BAKEOUT PROCEDURE FOR SMALL GLASS ULTRAHIGH VACUUM SYSTEMS

Fortunat Steinrisser

Abstract

A bakeout procedure for small glass ultrahigh vacuum systems is described which insures pressures well below 10^{-11} Torr. An optically dense zeolite trap and a valve were placed between diffusion pump and system. The trap was baked whenever it was loaded with gas, i.e., after glassblowing, system bakeout, and outgassing of the ion gauges. The valve between trap and system was kept closed during bakeout of the trap. During bakeout of the system, outgassing of the ion gauges, and regular operation of the system, the trap was refrigerated at liquid nitrogen temperature.

The observed partial pressures are given. Atmospheric He diffusing through the Pyrex glass and H_2 diffusing out of metal parts were the dominant residual gases. CO production during O_2 admission was small in comparison to processing without use of the isolation valve.

System Processing

In a recent communication by J. H. Singleton and W. J. Lange[1] it was reported that the main residual gas in their Pyrex glass systems of about two liters volume was CO_2. The lowest stable pressure was $\sim 5 \times 10^{-11}$ Torr when they processed their systems in the following way: a) trap refrigerated, system baked ~10 h at 420°C; b) trap isolated from system and pumped while baked at ~250°C; c) ion gauge outgassed by electron bombardment. It was observed that the lowest pressure was about one order of magnitude higher when between stages b) and c) the system was baked once more. Diffusion pumps giving an effective pumping speed of ~0.5 ℓ/sec at the systems were used with various pumping fluids.

The performance of some very similar systems (see Figure 1) has been examined in this laboratory during the past two years. They were made from Pyrex glass (Corning 7740) and had a volume of one to three liters. Usually a magnetic deflection type mass spectrometer[2] was included for partial pressure measurements. Bayard-Alpert gauges and Schuemann photocurrent suppressor gauges[3] were used for total pressure measurements. An optically dense zeolite trap filled with approximately 10 g of Molecular Sieve (Linde 13 X) could be refrigerated at liquid nitrogen temperature. A one inch valve served to isolate the trap from the system. Two-stage fractionating oil diffusion pumps (CVC GF-20) were used with Monsanto OS-124 oil; the pump was air cooled, and the pumping speed for N_2 at the system was ~0.5 ℓ/sec.

With the following procedures, pressures below 1×10^{-11} Torr were regularly obtained 2-3 days after exposing the system to atmospheric pressure: a) The system was pumped with a forepump to $\sim 10^{-3}$ Torr (the valve between system and diffusion pump was kept closed with the diffusion pump always running). Then the valve was opened and the system pumped for several hours with the diffusion pump. b) The trap was valved off from the system and baked at $\sim 350^{\circ}$C for four hours; the glass tubing between valve and trap and the valve were kept at $\sim 150^{\circ}$C to prevent oil condensation. c) The valve was opened after the trap had been refrigerated to liquid nitrogen temperature. Then the system was baked at 350°C for ~ 10 hours. d) Stage b) was repeated. e) The ion gauges were outgassed at 80 W for six hours. f) Stage b) was repeated. If necessary, the cycle c) to f) was repeated.

Partial Pressures

One of the systems was used for a detailed investigation of partial pressures during system processing. It was repeatedly cycled from atmospheric to very low pressure. It consisted of a Bayard-Alpert gauge WL-5966, a Schuemann photocurrent suppressor gauge of more recent design[4] with a low temperature filament, and a mass spectrometer.[2]

The main residual gas during bakeout of the system and outgassing of the gauges was CO; CO_2 was always less than CO. H_2 was also present and became the major residual gas when the system was close to room temperature.

To obtain low pressures, the gauges and mass spectrometer had to be outgassed at: 50 W (Bayard-Alpert), 120 W (Schuemann) with all metal parts except the filaments connected to the grid, 10 W (ion source of mass spectrometer). Pressures of less than 1×10^{-11} Torr were obtained two days after starting the processing. After three days, the system reached its final pressure in the low 10^{-12} Torr range as measured with the Schuemann gauge. These pressures are in nitrogen equivalent. A further decrease could be observed when the gauges were shut off. Table I gives the dominant partial pressures observed under different conditions. These pressures are actual pressures taking into account the sensitivity of the mass spectrometer for the different gases. Calibrations were made with the Bayard-Alpert gauge in the 10^{-9} Torr region.[5] From a paper by Davis[6] it is known that this mass spectrometer is linear down to the lowest pressures. Helium diffusing through the glass walls is the major component. H_2 is important, too, and very probably arises from the mass spectrometer source region as can be seen from an H_2 increase if the emission current is increased. Davis[6] reports a partial pressure of H_2 of 1 to 1.5×10^{-12} Torr due to outgassing of the mass spectrometer source. Our values are slightly higher because the source was operated at a higher emission current (.5 mA compared to .2).

TABLE I

Partial Pressures of Dominant Gases

Condition	H_2	He	CO
both gauges on	5.0×10^{-12}	8.0×10^{-12}	6.0×10^{-13}
Bayard-Alpert gauge off	4.0×10^{-12}	6.2×10^{-12}	6.0×10^{-13}
both gauges off	4.0×10^{-12}	5.3×10^{-12}	6.0×10^{-13}

The valve between system and pump was closed for eight days in an attempt to see how much gas was collected in the system. All filaments were off.

Table II gives the partial pressures after eight days: a) five min. after turning on the mass spectrometer with the valve closed; b) five min. after opening the valve; c) five hours after opening the valve. The He influx, Q, was calculated to 2.6×10^{-12} Torr ℓ/sec; from the relation $S = Q/P$ at equilibrium, the pumping speed S at $P = 5 \times 10^{-12}$ Torr was found to be $S \cong 0.5$ ℓ/sec. The H_2 evolution was much smaller when the mass spectrometer was off. This supports again the assumption that the heating of the mass spectrometer source by the hot filament is responsible to a large extent for the observed H_2 evolution. As one can see from a comparison of Table II with Table I, the system reached its base pressure again only a few hours after opening the valve.

TABLE II

Partial Pressures in Torr after Closing the Valve between System and Pump for 8 Days

Condition	H_2	He	CO
(a) Valve closed, mass spectrometer on for 5 min.	2.6×10^{-10}	6.6×10^{-7}	8.0×10^{-12}
(b) Valve opened for 5 min.	6.0×10^{-12}	5.4×10^{-12}	8.0×10^{-12}
(c) Valve opened for 5 h	3.4×10^{-12}	5.0×10^{-12}	6.0×10^{-13}

CO-Production during O_2 Admission

In another experiment, the influence of processing upon CO-production during O_2-admission was investigated. Some of the earlier experiments by Schuemann, Segovia and Alpert[7] were repeated. The main difference was the very small CO production rate observed in this experiment if the system was kept oil-free. It was found that it makes a big difference whether the valve and the glass tubing between valve and trap were kept at $\sim 150^{\circ}C$ or at room temperature during bakeout of the trap. In the latter case, there was apparently some oil condensation in the valve and the glass tubing. Oil cracking patterns could be seen immediately after turning on the low temperature filament in the mass spectrometer (Figure 2). Only 15 min. later, the typical oil cracking pattern had disappeared, and only H_2 and CO could be found (Figure 3) in large quantities.

The system still reached pressures in the low 10^{-11} Torr range. In this case, however, the CO pressure reached more than 20% of the O_2 pressure under equilibrium conditions.

When the processing was done as described earlier, i.e. if valve and glass tubing between valve and trap were kept at $\sim 150^{\circ}C$ during bakeout of the trap, the CO pressure was only around 2% of the O_2 pressure under identical conditions. One regular filament in the Bayard-Alpert gauge was replaced by an ultra-pure W filament. With this filament, even lower CO production was observed. In Table III, the CO pressure in percent of O_2 pressure is given under different conditions and for times T = 5 min. and T = 1 day after O_2 admission.

All of our observations are in agreement with results found by Becker, Becker and Brandes[8] on "Reactions of Oxygen with Pure Tungsten and Tungsten Containing Carbon." Carbon from oil cracking products apparently diffuses into the W filaments. In an oxygen atmosphere, CO is formed on the hot tungsten filament and carbon diffuses out again.

TABLE III

CO Production (in % of O_2, $P_{O_2} \approx 5 \times 10^{-7}$ Torr)

Emission Currents: Mass Spectrometer--1 mA; B.A. Gauge--10 mA

Conditions	% CO (T = 5 min)	% CO (T = 1 day)
Only mass spectrometer on, low temperature filament, no oil	.75	.4
Only mass spectrometer on, W filament, no oil	2.0	.9
B.A. gauge on, regular filament, no oil	2.5	2.1
B.A. gauge on, ultra-pure filament, no oil	1.8	.4
B.A. gauge on, regular filament, with oil	3.0	25.0

Conclusions

1) Small glass ultrahigh vacuum systems with a zeolite trap between diffusion pump and system are capable of pressures in the low 10^{-12} Torr range (nitrogen equivalent).

2) A valve between trap and system is necessary for system processing.

3) With the technique described in this note, pressures below 10^{-11} Torr may be obtained two days after opening the system to air.

4) Bakeout temperatures of 350°C are sufficient for glass systems.

5) CO production in the presence of oxygen and a hot filament can be greatly reduced by this technique.

Acknowledgments

The author would like to thank D. Alpert, A. Dallos, R. N. Peacock, and F. M. Propst for helpful discussions, and W. I. Lawrence for excellent glassblowing work.

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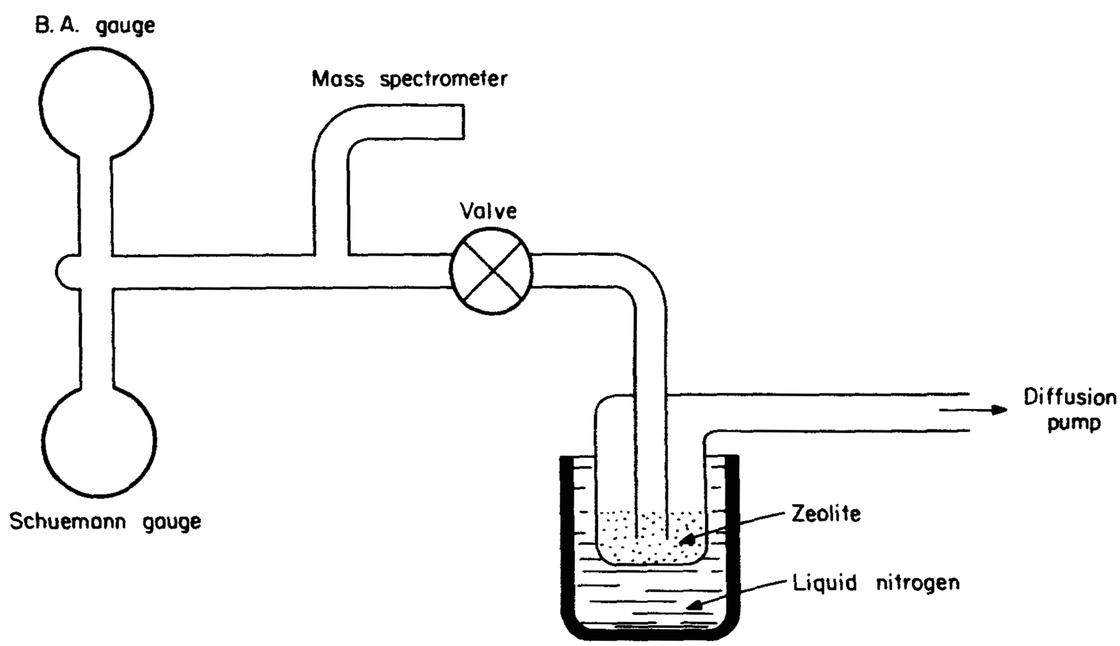

Fig. 1. A schematic view of the vacuum system.

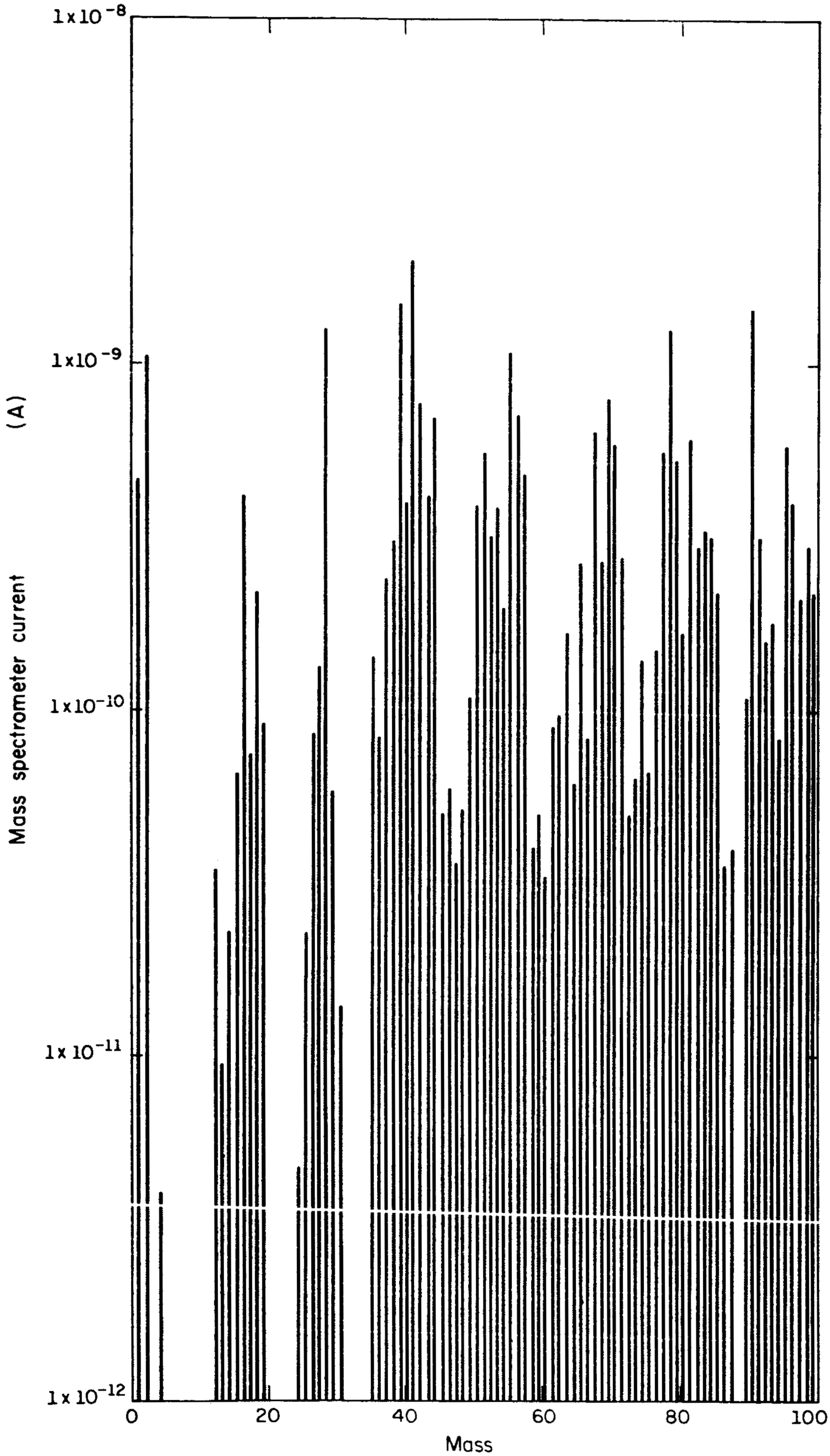

Fig. 2. Mass spectrum with characteristic oil cracking pattern taken immediately after turning on the low temperature filament in the mass spectrometer.

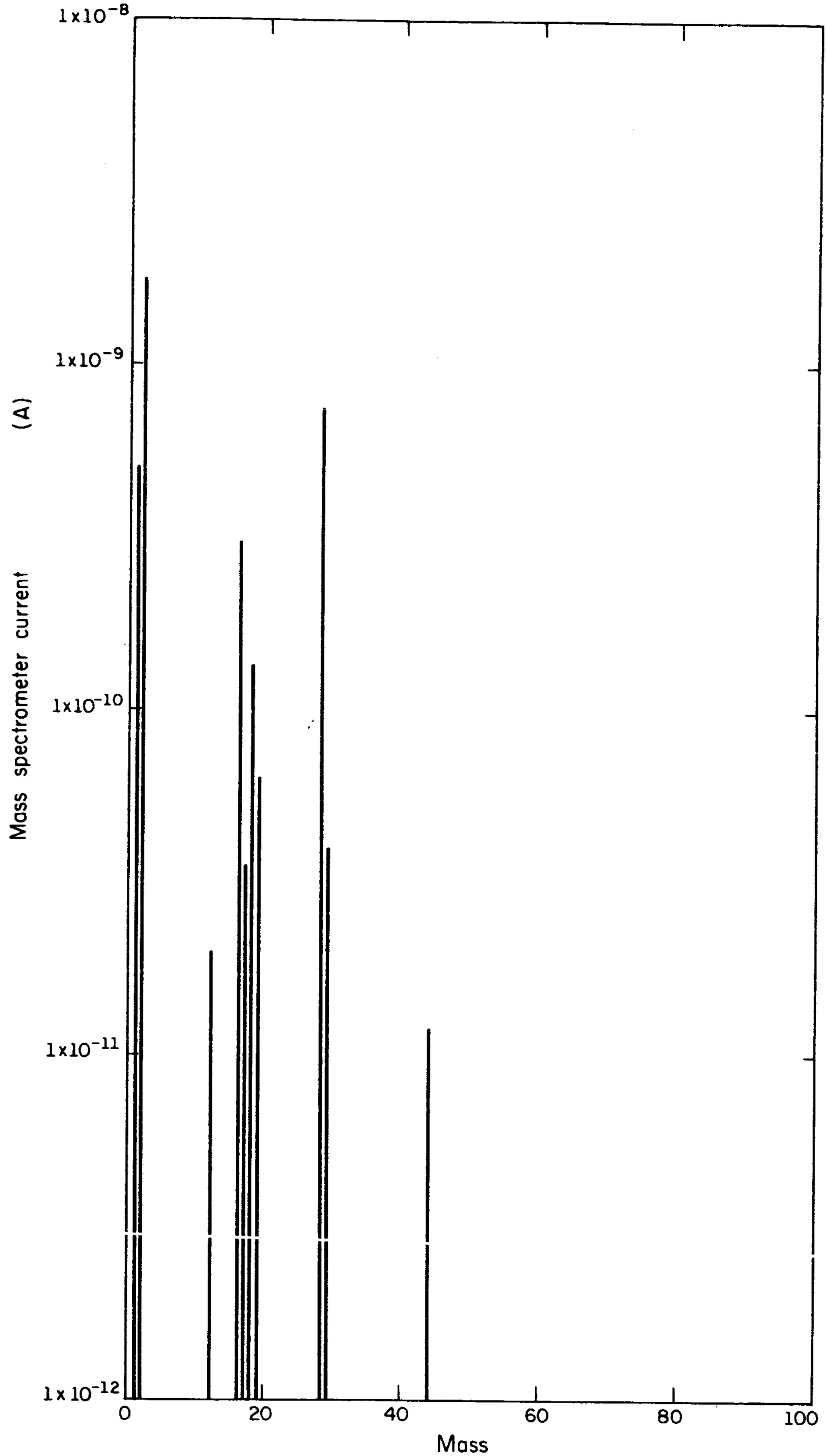

Fig. 3. Same mass spectrum as in Fig. 2, but with the filament on for 20 min. Note that only hydrogen and CO are left in large quantities. The oil cracking pattern has practically disappeared.

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4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

5. AUTHOR(S) (Last name, first name, initial)

Steinrisser, Fortunat

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS.
June, 1966	12	8

8a. CONTRACT OR GRANT NO. DA 28 043 AMC 00073(E) b. PROJECT NO. 20014501B31F c. Also National Aeronautics and Space Administration under Grant d. NASA NsG-376.	9a. ORIGINATOR'S REPORT NUMBER(S) R-299 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

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DD FORM 1473 1 JAN 64

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